IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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12/4/3

Applicant: Mark Mattox

Title: ***METHOD TO DECREASE IRON SULFIDE DEPOSITS IN PIPE LINES***

Appl. No.: 10/044,767 Filing Date: 01/10/2002

Examiner: A. Markoff Art Unit: 1746

DECLARATION UNDER 37 C.F.R. § 1.132

Commissioner for Patents
PO Box 1450
Alexandria, Virginia 22313-1450

I, Edward J. Valente, hereby declare that:

1. I am a scientific consultant for Synergy Chemicals, Inc., the assignee of the captioned application.

2. I am a full professor of chemistry at Mississippi College. I have conducted research in the fields of organic and physical chemistry for 25 years. My research includes the study of coordination compounds of transition metals, including iron. I have published over 100 papers in these areas. My qualifications are set out in my *curriculum vitae*, which is attached hereto as APPENDIX A.

3. I have reviewed and understood the subject patent application. I have also read and understood the final Office Action dated April 1, 2003, the Advisory Action dated August 25, 2003, and cited U.S. patent No. 6,517,617 to Chartier *et al.*

4. As the experimental data set forth below, the presence of oxygen in a composition containing the components water, a soluble ammonium salt, and tetrakis(hydroxylmethyl) sulfate ("THPS") would render the composition incapable of complexing iron (II) compounds such as iron sulfide. The data thus supports my expert opinion that foams containing these components and 90 – 95 vol. % (v/v), as is the case for the foams disclosed by Chartier, cannot complex iron sulfide.

5. A control composition ("the control"), which corresponds to the composition recited in the method claims at issue, was prepared by dissolving THPS (12 wt. %) and ammonium chloride (0.8 wt %) in deionized water. An identical composition ("the test") was also prepared for comparative purposes.

6. Air was sparged rapidly into the test composition (~ 500 mL/min) during 24 hours. The test composition thus approximates the foams (90 – 95 vol. % of air) that are disclosed by Chartier. Over the same 24 hour period, control composition was prevented from contacting air.

7. Two sets of experiments evaluated the effects of the presence of oxygen in the test composition. The first set of experiments identified the evolution of phosphorus-containing compounds in each composition. The second set of experiments evaluated the abilities of these compositions to complex iron (II).

8. In the first set of experiments, the control and test compositions were separately monitored by $^{31}P\{H\}$ NMR during the 24 hour period mentioned above.

9. As Table 1 (below) shows, the **control** composition, at all times during this evaluation, contained three phosphorus-containing compounds in the following relative concentrations:

Table 1. Control Composition in Absence of Oxygen

Compound	Relative Concentration (%)
unreacted THPS	19.4
tris(hydroxymethyl)phosphine	75.8
trishydroxymethylphosphine oxide	4.8

10. Of these three phosphorus-containing species, only trishydroxymethylphosphine ("TRIS") is responsible for giving rise to the complexation of iron (II) compounds, specifically when TRIS is reacted with a soluble ammonium salt such as ammonium chloride in the presence of iron (II). THPS and trishydroxymethylphosphine oxide ("TRIS oxide"), by contrast, are incapable of complexing iron (II). Thus, as shown explicitly by the second set of experiments described below, the control composition, at all times, contained the prerequisite compounds – TRIS and ammonium chloride – that are necessary for complexing iron (II) compounds such as iron sulfide. (The presence of a small amount of TRIS oxide is ascribed to the presence of trace amounts of oxygen in the deionized water that was used to prepare the control composition.)

11. The **test** composition, by contrast, decomposed markedly over the 24 hour period to leave exclusively the two species, namely THPS and TRIS oxide, that do not give rise to iron (II) complexation (Table 2):

Table 2: Exposure of Test Composition to Oxygen

Compound	Relative Concentrations (%) at time t (h)		
	$t = 1$	$t = 3$	$t = 24$
unreacted THPS	21.6	29.5	33.3
tris(hydroxymethyl)phosphine	71.2	49.7	0.0
trishydroxymethylphosphine oxide	7.2	20.8	66.7

12. The results of the first set of experiments thus demonstrate that a composition of THPS, ammonium chloride, water, and oxygen results in a chemically much different composition than one that is not exposed to oxygen. Specifically in the case of the **test** composition, TRIS is completely decomposed (i.e., oxidized), thereby rendering this composition incapable of complexing iron (II) as the data below shows.

13. The second set of experiments evaluated the abilities of the control and test compositions to complex iron (II). Portions of each composition (2 mL) were combined with 1 mL of a solution of iron (II) sulfate (100 mg of iron(II)sulfate heptahydrate in 10 mL deionized water), diluted with 1 mL water, and then monitored over time by visible spectroscopy at 500 nm.

14. A composition of THPS, water, and ammonium chloride reacts with virtually any convenient source of iron (II), including iron (II) sulfide and iron (II) sulfate, to invariably give the same iron (II) complex, which exhibits an absorption maximum at 500 nm. Thus, the intensity at 500 nm correlates directly with the complexation of iron (II). (For the convenience of obtaining accurate intensity measurements, iron (II) sulfate was employed in these experiments because iron (II) sulfide is insoluble in water. Independent tests employing iron sulfide give the identical end results.)

15. The **control** composition, when contacted with iron (II) sulfate, gave rise to 0.8 absorbance units (au) within 20 seconds, indicating the immediate complexation of iron (II). After 4 minutes, the absorbance had not changed, thus signaling that iron (II) complexation was complete after 20 seconds.

16 By contrast, aliquots of the air-sparged **test** compositions, when similarly treated with iron (II) sulfate, exhibited little to no iron (II) complexation (Table 3):

Table 3. Iron (II) Complexation by Test Composition

Time (h) that Composition Was Sparged with Air (h)	Rate of Iron (II) Complexation (au) After Time *t*
4	0.13 au (20 s)
	0.27 au (4 min.)
24	0.0 au (20 min.)
	0.0 au (4 min.)

17. The results in Table 3 demonstrate that the presence of oxygen in a composition of THPS, ammonium, chloride, and water renders the composition incapable of complexing iron (II).

18. The foams disclosed by Chartier contain 90 – 95 vol. % air, which is a much higher concentration of air than was present in the air-sparged test compositions described above. The presence of a greater concentration of air, and thus a higher concentration of oxygen, ensures that THPS, ammonium chloride, and water would undergo more rapid decomposition, *via* oxidation of TRIS, such as evidenced in Table 1 above.

19. Additionally, the foam of Chartier enforces a much higher surface area contact between air and the THPS, ammonium chloride, and water components than that achieved by simply sparging air through a solution of the same components. Greater surface area contact between the key reactants here –oxygen and TRIS – ensures greater rates and degrees of oxidation to TRIS oxide, which does not give rise to iron (II) complexation.

20. The foam that is explicitly recited in claim 1 of Chartier contains only 2 – 3 wt. % of THPS, whereas the control and test compositions contained 12 wt. % THPS. A lower concentration of THPS relative to the concentration of oxygen would ensure a more rapid and complete decomposition of a composition containing THPS, ammonium chloride, and water.

21. These features of the disclosed foam, viewed particularly in light of the results collected in Table 3, renders it a virtual certainty that the oxygenated foams of Chartier do not complex iron sulfide.

22. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application of any patent issuing thereon.

Respectfully submitted,

Date November 6, 2003 By _____

Prof. Edward J. Valente

APPENDIX A
Curriculum Vitae

Edward Joseph Valente

Born: January 16, 1949, in San Francisco, CA

Education: Ph.D. (Chemistry) University of Washington, Seattle; March, 1977

B.S. (Chemistry) University of California, Berkeley; March, 1971

Positions: Professor of Chemistry, Mississippi College, Clinton, MS; 1989-present.

Associate Professor of Chemistry, Mississippi College, Clinton, MS; 1983-1989.

Adjunct Professor of Chemistry, Jackson State University, Jackson, MS; 1988-present.

Visiting Senior Research Scientist, Smith, Kline & French R & D, King of Prussia, PA; Summer, 1988.

Post-doctoral Scholar, Department of Chemistry, University of Michigan, Ann Arbor, MI; 1981-1983.

Instructor in Chemistry, Coastal Carolina Community College, Jacksonville, NC; 1979-1981.

Post-doctoral Research Associate; Department of Chemistry, University of North Carolina, Chapel Hill, NC; 1978-1979, Summer, 1980.

Post-doctoral Research Assistant; Department of Pharmaceutical Sciences, University of Southern California, Los Angeles, CA; 1977-1978.

Professional Organizations:

American Chemical Society (1976-present)

American Crystallographic Association (1980-present)

Mississippi Academy of Sciences (1983-present; Chair, Chemistry, 1989-91, 1998)

Honors: Chemist of the Year American Chemical Society, Mississippi Section, 1998-9

Distinguished Lecturer; School of Arts and Sciences, Mississippi College, 1998

Professor of the Year, Mississippi College, 2003-4

Teaching: Organic Chemistry, Medicinal Chemistry, General Chemistry, Instrumental Analysis

Research Interests:

Synthesis, spectroscopy, and structure of derivatives of 4-hydroxy-coumarin related to warfarin.

Nucleation in nozzle flow; electron diffraction studies of liquids and microcrystalline solids.

Structures of organic diastereomeric salts, aspects of molecular recognition.

Modulation of polar bond lengths and correlation with organic reactivity.

Grants

Dreyfus & Keck Foundations: A Modern Magnetic Resonance Spectroscopy for an Enhanced Chemistry Curriculum and Support of Local Two-Year College Organic Chemistry, 3/01-3/03.

National Science Foundation (CCLI -A&I), AModern Magnetic Resonance Spectroscopy for an Enhanced Chemistry Curriculum and Support of Local Two-Year College Organic Chemistry@, 3/01-3/03.

National Science Foundation (CCLI-A&I), Washington, DC; ASpectroscopy for Student-Designed General Chemistry Explorations and Advanced Chemistry, 9/99-8/01.

National Science Foundation (ILI), Washington, DC; ALuminescence, Scintillation and Spectrofluorimetry for Analytical and Bioanalytical Laboratories, 7/96-6/98.

Office of Naval Research, Bethesda, MD; Structure Property Relations (With J. D. Zubkowski, Jackson State University) 6/88-5/91;6/91-5/94; 6/94-5/97; renewal (6/97-5/99); renewal (6/99-5/01).

National Institutes of Health, Div. of Research Resources, Bethesda, MD, (AREA) "Xray Diffraction Support for Undergrad. Research"; 4/90 - 3/92.

National Science Foundation (ILI), Washington, DC; "Gas Chromatograph-Mass Spectrometry for a Modern Chemistry Curriculum"; 7/89 - 6/91.

National Science Foundation (ILI), Washington, DC; "Fourier-Transform Infrared Spectroscopy for a Modern Chemistry Curriculum"; 9/88 - 8/90.

American Heart Association, Mississippi Affiliate, Jackson, MS; "Anti-coagulants as Probes of Binding to Macromolecules", 7/84 - 6/87.

Research Corporation, Tucson, AZ; "Stability and Chemistry of Hexachloroarsenate (V) Salts", 6/84 - 6/85.

Southern Regional Education Board, Atlanta, GA; " Structure of Methyl Ketals of Warfarin and Related Compounds", 3/80.

Attachment **Publications**

Dissertation: "A Structural Study of Coumarin Anticoagulants and Other Derivatives of 4-Hydroxycoumarin", University of Washington, 1977. Advisors: E. C. Lingafelter (chemistry) & W. F. Trager (pharmaceutical sciences).

1. Crystal and Molecular Structure of Warfarin, a Clinical Oral Anticoagulant Drug. E. J. Valente, W. F. Trager & L. H. Jensen. Paper delivered before the American Crystallographic Association Meeting, March, 1974.

1a. Crystal and Molecular Structure and Absolute Configuration of (-)(S)-Warfarin. **E. J. Valente**, W. F. Trager & L. H. Jensen, *Acta Cryst.* **B31**, 954-960 (1975).

2. (-)-3-(1-Phenylpropyl)-4-hydroxycoumarin. **E. J. Valente**, W. F. Trager & E. C. Lingafelter, *Acta Cryst.* **B32**, 277-279 (1976).

3. The Structure of Warfarin in Solution. **E. J. Valente**, E. C. Lingafelter, W. R. Porter & W. F. Trager, *J. Med. Chem.* **20**, 1489-1493 (1977).

4. The Conformation of Selected 3-Substituted 4-Hydroxycoumarins in Solution by NMR: Warfarin and Phenprocoumon. **E. J. Valente**, W. R. Porter & W. F. Trager, *J. Med. Chem.* **21**, 231-234 (1978).

5. The Anomalous Chiroptical Properties of Warfarin and Phenprocoumon. **E. J. Valente** & W. F. Trager, *J. Med. Chem.* **21**, 141-143 (1978).

6. Conformation of Dihydropyran Rings: The Structures of Two 3,4-Dihydro-2**H**,5**H**-pyrano[3,2-c][1]benzopyran-5-ones. **E. J. Valente**, B. D. Santarsiero & V. Schomaker, *J. Org. Chem.* **44**, 798-802 (1979).

7. Proton Magnetic Resonance Study of the In Vitro Decomposition of 4-Hydroxy-cyclophosphamide, a Microsomal Metabolite of Cyclophosphamide. E. J. Valente, K. K. Chan & K. L. Servis, Paper presented before the Academy of Pharmaceutical Sciences and Medicinal Chemistry, April, 1979.

7a. Proton Magnetic Resonance Studies of the Decomposition of 4-Hydroxycyclophos-phamide, a Microsomal Metabolite of Cyclophosphamide. **E. J. Valente**, K. K. Chan & K. L. Servis, *J. Pharm. Biomed. Sci.* **2**, 89-92 (1984).

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9. NMR Configuration Correlation of Amine Derivatives of χ-Methyl-χ-methoxypenta-fluorophenylacetic Acid. L. R. Pohl, **E. J. Valente** & W. F. Trager, *J. Org. Chem.* **45**, 543-546 (1980).

10. The Synthesis of C-2 Isotopically Labelled Optically Pure Warfarin and Phen-procoumon. W. R. Porter, K. Kunze, **E. J. Valente** & W. F. Trager, *J. Labelled Compounds and Radiopharm.* **17**, 763-773 (1980).

11. 2-Methyl-2-hydroxy-4-cyclohexyl-3,4-dihydro-2**H**,5**H**-pyrano[3,2-c][1]benzo-pyran-5-one. **E. J. Valente** & D. J. Hodgson, *Acta Cryst.* **B35**, 3099-3101 (1980).

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15. Electron Diffraction Studies of Supersonic Jets. Paper before the IX Austin Symposium on Molecular Structure. Austin, TX. R. K. Heenan, **E. J. Valente** & L. S. Bartell, March, 1982

15a. Electron Diffraction Studies of Supersonic Jets, II: Formation of Benzene Clusters. R. K. Heenan, **E. J. Valente** & L. S. Bartell, *J. Chem. Phys.* **78**, 243-248 (1983).

16. Electron Diffraction Studies of Supersonic Jets, V: Low Temperature Crystalline Forms of SF_6, SeF_6, TeF_6. **E. J. Valente** & L. S. Bartell, *J. Chem. Phys.* **79**, 2683-2685 (1983).

17. Electron Diffraction Studies of Supersonic Jets, VI: Microdrops of Benzene. **E. J. Valente** & L. S. Bartell, J. *Chem. Phys.* **80**, 1451-1457 (1984).

18. Electron Diffraction Studies of Supersonic Jets, VII: Liquid and Crystalline Carbon Tetrachloride. **E. J. Valente** & L. S. Bartell, J. *Chem. Phys.* **80**, 1458-1461 (1984).

19. Structure of Dihydropyran Rings. Paper before the American Chemical Society Meeting, Seattle, WA. **E. J. Valente**, L. S. Bartell & V. Schomaker, March, 1983.

19a. A Structure containing Diastereomers, (2S,2R)-trans- and (2R,4R)-cis-2-Hydroxy-2,4-dimethyl-3,4-dihydro-2**H**,**5H**-pyrano[3,2-c][1]benzopyran-5-one. **E. J. Valente** & V. Schomaker. *Acta Cryst.* **C40**, 1068-1070 (1984) and *Proc. Miss. Acad. Sci.* (Suppl.) **29**, 14 (1984).

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40a. Discrimination in Resolving Systems: Ephedrine Halomandelates. E. J. Valente, Jeffrey Zubkowski, and Drake S. Eggleston. A paper presented before the annual meeting of the American Crystallographic Association, Pittsburgh PA, August, 1992.

40b. Structures and Properties of Three Ephedrinium Halomandelates. Chris Miller & **E. J. Valente** and Jeff Zubkowski. A paper presented before the 57th annual meeting of the Mississippi Academy of Sciences, Jackson MS, February, 1993.

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